PCT WORLD INTELLECTUAL PROPERTY ORGANIZATION
International Bureau

INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification 6 :
G01N 3/42

A1

(11) International Publication Number: WO 99/45361

(43) International Publication Date: 10 September 1999 (10.09.99)

(21) International Application Number: PCT/GB99/00673

(22) International Filing Date: 8 March 1999 (08.03.99)

(30) Priority Data:
9804799.6 7 March 1998 (07.03.98) GB

(71) Applicant *(for all designated States except US)*: COVENTRY UNIVERSITY [GB/GB]; Priory Street, Coventry CV1 5FB (GB).

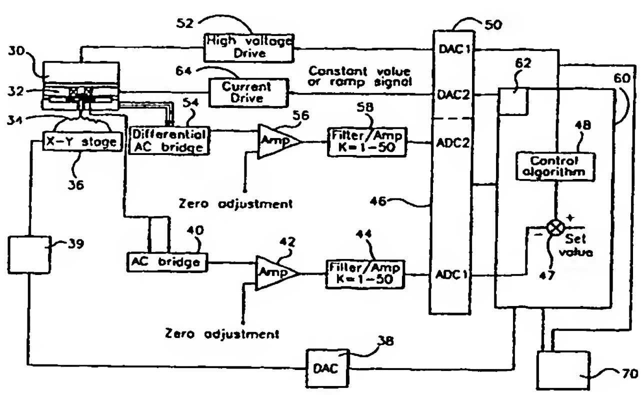
(72) Inventor; and
(75) Inventor/Applicant *(for US only)*: LIU, Xianping [CN/GB]; Coventry University, Priory Street, Coventry CV1 5FB (GB).

(74) Agent: HALLAM, Arnold, Vincent; Lewis & Taylor, 5 The Quadrant, Coventry CV1 2EL (GB).

(81) Designated States: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SL, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).

Published
With international search report.

(54) Title: NANOTRIBOLOGICAL PROBE MICROSCOPE



(57) Abstract

A scanning probe microscope for measuring the topography, local hardness and frictional force of an object surface has a stage (36) for mounting the object, a probe mounted relative to the stage and a z positioner (30) for moving the probe and the stage (36) relative to one another along a first, z axis and in an x-y plane orthogonal to the first, z axis. The probe has a body and a stylus (12) for contacting the object surface which is resiliently mounted to the body so as to allow movement of the stylus relative to the body along the z axis. An electromagnetic actuator (32) applies a force to the stylus to bias the stylus relative to the body along the z axis towards the stage (36). A capacitative sensor (25) monitors the position and tilt of the stylus (12) relative to the body along the z axis and provides a position signal representative of the stylus position on the z axis. A control circuit includes a feedback circuit connecting the z positioner (30) and the capacitative sensors (25) to control the z positioner (30) to bring the stylus (12) and the probe into a preselected relationship along the z axis in response to initial contact of the stylus with the object surface. By applying force to the stylus along the z axis at various points of contact on the object the surface topography and hardness can be measured. By dragging the stylus across the surface and measuring the tilt of the stylus the friction of the surface can be measured.

FOR THE PURPOSES OF INFORMATION ONLY

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AL	Albania	**ES**	Spain	**LS**	Lesotho	**SI**	Slovenia
AM	Armenia	**FI**	Finland	**LT**	Lithuania	**SK**	Slovakia
AT	Austria	**FR**	France	**LU**	Luxembourg	**SN**	Senegal
AU	Australia	**GA**	Gabon	**LV**	Latvia	**SZ**	Swaziland
AZ	Azerbaijan	**GB**	United Kingdom	**MC**	Monaco	**TD**	Chad
BA	Bosnia and Herzegovina	**GE**	Georgia	**MD**	Republic of Moldova	**TG**	Togo
BB	Barbados	**GH**	Ghana	**MG**	Madagascar	**TJ**	Tajikistan
BE	Belgium	**GN**	Guinea	**MK**	The former Yugoslav Republic of Macedonia	**TM**	Turkmenistan
BF	Burkina Faso	**GR**	Greece	**ML**	Mali	**TR**	Turkey
BG	Bulgaria	**HU**	Hungary	**MN**	Mongolia	**TT**	Trinidad and Tobago
BJ	Benin	**IE**	Ireland	**MR**	Mauritania	**UA**	Ukraine
BR	Brazil	**IL**	Israel	**MW**	Malawi	**UG**	Uganda
BY	Belarus	**IS**	Iceland	**MX**	Mexico	**US**	United States of America
CA	Canada	**IT**	Italy	**NE**	Niger	**UZ**	Uzbekistan
CF	Central African Republic	**JP**	Japan	**NL**	Netherlands	**VN**	Viet Nam
CG	Congo	**KE**	Kenya	**NO**	Norway	**YU**	Yugoslavia
CH	Switzerland	**KG**	Kyrgyzstan	**NZ**	New Zealand	**ZW**	Zimbabwe
CI	Côte d'Ivoire	**KP**	Democratic People's Republic of Korea	**PL**	Poland		
CM	Cameroon	**KR**	Republic of Korea	**PT**	Portugal		
CN	China	**KZ**	Kazakstan	**RO**	Romania		
CU	Cuba	**LC**	Saint Lucia	**RU**	Russian Federation		
CZ	Czech Republic	**LI**	Liechtenstein	**SD**	Sudan		
DE	Germany	**LK**	Sri Lanka	**SE**	Sweden		
DK	Denmark	**LR**	Liberia	**SG**	Singapore		
EE	Estonia						

Title: **Nanotribological Probe Microscope**

Background of the Invention

Field of the Invention

Nanotribology, the frictional interaction of small areas of surfaces in contact is commanding more attention than ever in research fields ranging from basic physics to industrial production. This is due to the rapid development of Nanotechnology and Micromachining technology in applications such as microsensor, microactuators, high density memory devices, micromotors and micropumps. Frictional interaction results in wear and is at present, and is likely to remain, the limiter of performance of those microdevices. Stress and stress distribution in a thin film dominates the performance of micro-sensors and actuators fabricated by surface micromachining techniques. On the one hand in these precision miniature mechanical systems, the frictional force does not scale down with dimension and sometimes even increases dramatically. On the other hand it is possible that by trial and error a near perfect lubricating condition can be achieved.

Friction, lubrication, adhesion, and fracture processes are controlled by the mechanical response of two contact surface materials. In contrast to conventional tribology where frictional interaction of solid surfaces is dominated by the properties of bulk materials, under a light load and small masses, the physical and chemical properties of the surfaces are important in microtribology and nanotribology. It has been reported that the friction and wear behaviour of ceramics and ceramic coatings are influenced by the environment in which components operate. There is thus great interest in being able to measure directly the elastic and inelastic responses of material on this scale.

Thin film lubrication has been widely used in precision translations, high density memory drives and integrated silicon micromechanisms to improve performances through the reduction of friction and wear at the contact surfaces. In recent years the electrodeposition

of conductive polymers onto well defined surfaces has attracted more attention in microengineering and nanotechnology. A distinctive advantage of this technique is that the deposition process is well-controlled and works on irregular surfaces. The choice of monomer, counter-ion, solvent, and growth potential determines the film morphology and can lead to smooth, fibrillar or spherical microstructures which may enable the optimisation of a polymer coating to a particular tribological requirement. However, it has been found that films produced under a nominal same condition gives variable performance in friction which adds complexity in data interpretation and the control of the film production. Experimental evidence shows that the film morphology such formed varies in the molecule size and shape and the distribution which may affect its mechanical and tribological properties. Therefore, to characterise such a surface, it is necessary to correlate the three aspects of surface: morphology, mechanical response and frictional behaviour by direct measurements on the same surface point.

In addition to the materials interest, scanning probe microscopes also rely on the mechanical response of small areas of surfaces, particularly contact mode AFMs (atomic force microscopes). The elastic deformation of the tip-sample system during AFM measurements falsifies the topography and causes incorrect values for interaction force as well as the loss of true atomic resolution.

Scanning probe microscopy (SPM) is a global term for a multitude of microscopy modes, all based on the same principle. Scanning microscopes image by "touch".

Description of the Prior Art

Scanning microscopy uses a cantilever arm mounted to the tip of which is a triangular probe whose apex is only tens of nanometres wide. A laser beam is used to monitor movement of the end of the cantilever arm as the probe is moved across a surface being imaged. The movement of the end of the cantilever arm is translated by a computer into three-dimensional information.

Scanning probe microscopy (SPM) such as scanning tunnelling microscope (STM) and atomic force microscope (AFM) is a global term for imaging a surface via a sharp tip to reveal surface structure down to atomic levels. Since their inventions, variants of STM/AFM have been developed to extend the measurement of surface topography to other surface related phenomena. Attempts have been made successfully by using an AFM or its equivalent force probe microscopes to monitor surface hardness or elastic modulus, or interfacial force, or frictional force together with the surface topography, but none of them were able to measure the above three functions together. Most of AFMs and the variants to date only control and monitor displacements and forces are inferred from a nominal known spring constant of a cantilever. This can be a problem when the tip-sample interaction is significant, thus the contact stiffness dominates the spring behaviour of the cantilever which leads to the inferred force value worthless. Modifications have been made by some researchers by introducing force instead of displacement into the feedback control loop of AFM, but their performances are limited due to the fact that these modifications are based on the commercial AFMs which were designed primarily for surface imaging. The main limitation of these commercial instruments is the limit range for force application, which will not cope with the increasing demand for normal load up to mN region to cover a wide range of materials for investigation of friction, indentation, wear and fatigue.

Brief Summary of the Invention

The present invention seeks to provide an improved scanning probe microscope.

Accordingly, the present invention provides a scanning probe microscope for measuring the hardness of a surface of a sample object at a location on said surface, comprising:

a stage means for mounting the object;

a probe mounted relative to said stage means;

first drive means for moving the probe and the stage means relative to one another along a

first, z axis and in an x - y plane orthogonal to said first, z axis;

wherein the probe has:

a body;

stylus means for contacting the object surface;

mounting means resiliently mounting the stylus to the body so as to allow movement of the stylus relative to the body along the z axis;

actuator means for moving the stylus means relative to the body along the z axis towards the stage means;

and position monitoring means for monitoring the position of the stylus relative to the body along the z axis and providing a position signal representative thereof;

and wherein the microscope further comprises:

and a control circuit including a feedback circuit connecting the first drive means and the position monitoring means and operable for controlling the first drive means to bring the stylus and the probe into a preselected relationship along the z axis in response to initial contact of the stylus with the object surface;

and said control circuit further comprises second drive means for actuating said actuator means to apply a preselected force to the stylus means subsequent to said initial contact, and signal means for receiving said position signal and providing a hardness signal representative of the local hardness of the object surface at the point of contact of the stylus with the surface.

The present invention also provides a method of measuring the local hardness of an object

surface using a scanning probe microscope having a probe comprising:

a body;

stylus means for contacting the object surface;

mounting means resiliently mounting the stylus to the body so as to allow movement of the stylus relative to the body along the z axis;

actuator means for moving the stylus means relative to the body along the z axis towards the stage means;

and position monitoring means for monitoring the position of the stylus relative to the body along the z axis and providing a position signal representative thereof;

wherein the method comprises the steps of:

a) monitoring the position of the stylus relative to the probe body in the z axis;

b) moving the probe towards the surface along the z axis until a change in the position of the stylus relative to the body indicates contact of the stylus at a selected point on the surface;

c) moving the probe along the z axis relative to the stylus whilst maintaining contact of the stylus with the object surface until a preselected relationship is established between the positions of the stylus and the body along the z axis to set a datum position for the stylus at said selected contact point;

d) increasing by a preselected amount the force applied to the stylus along the z axis to move the stylus into the object surface;

e) monitoring the movement of the stylus in the z direction relative to the datum and

generating a position signal in dependence thereon;

f) and processing said position signal to provide a hardness signal representative of the hardness of the object surface at said selected surface contact point.

The present invention is further described hereinafter, by way of example, with reference to the accompanying drawings, in which:

Figure 1 is a schematic side elevation of a probe of a preferred embodiment of nanotribological probe microscope (NPM) according to the present invention;

Figure 2 is a sectional plan view along the line 2-2 of Figure 1;

Figure 3 is a block circuit diagram of the preferred form of NPM according to the present invention;

Figure 4 is a flow chart illustrating the operational steps of the microscope of Figure 3.

Referring firstly to Figures 1 and 2 a probe 10 of a preferred form of nanotribological probe microscope (NPM) has a body 11 and a diamond stylus 12 (Berkovich tip) mounted on a silica rod 14 which is a few millimetres long. The rod is fixed to the centre of a thin cross-shaped cantilever or beam 16 so as to be suspended by the beam 16 which is secured to the body at each of its four ends 16a, b, c, d and acts as a flexible spring. This allows vertical movement of the stylus 12, i.e. along the axis of the rod 14 and also allows some torsional movement of the beam 16. The stylus is thus compliantly coupled to the body. Secured to the beam 16, on the opposite side to the stylus 12, is a permanent magnet 18. This is surrounded by a coil 20. The coil 20 and magnet 18 form a force actuator 32 which enables the application of a force to the stylus 12 by passing current through the coil 20. The magnitude of the force applied to a sample surface 34 (Figure 3) via the stylus can be adjusted by controlling the current in the coil.

Four electrodes 22 are positioned each below a respective arm of the beam 16. These electrodes are conveniently deposited on a top mounting plate 24 which has an etched depth of about 50 μm for the four electrodes 22. The cross-beam 16 is made from 25 μm or 50 μm thick foil of Cu/Be and may be used as a common electrode to provide four capacitive sensors 25 with the electrodes 22. Alternatively, as shown a thin silica wafer 26 may be secured to the beam 16 to form the common electrode. The wafer 26 is more rigid than the beam 16 and can be better aligned parallel with the electrodes 22.

The four individual capacitive sensors 25 are configured as two pairs of capacitive sensors. One pair serves as a height/force sensor for measuring the vertical movement of the stylus and hence the topography of the sample 34 and the axial force applied to the stylus or the force normal to the surface 34 at the stylus 12. This pair of capacitative sensors can thus be used to measure the force which is due to the deflection of the beam 16 and is added to or subtracted from the normal force at the stylus 12. The second pair of the capacitive sensors 25 is a friction sensor operated in a differential mode for measuring the torsional motion of the beam 16 and thus the frictional force at the stylus 12.

The NPM is operated in a similar manner to a normal contact mode AFM but under a controlled loading force by the combination of the magnet 18/coil 20 and the beam 16. It measures surface topography up to 10 μm with subnanometre resolution, force application in a range from 10^{-8} N to 10^{-3} N or 10^{-2} N for elastic modulus mapping and lateral force measurement in a similar force range resolving to around 10 nN for frictional measurement.

Because the probe comprises mainly the stylus 12, two pairs of capacitive sensors 25 and the force actuator of the coil 20 and magnet 18 it can be made small and compact in size and can be employed in any commercial x - y - z stage for three dimensional measurements or x - y positioner for two dimensional measurements.

The x - y movements of the probe 10 can be produced by the NPM via a PZT stage 28, in which case the whole probe is assembled in a solid shielding form to improve thermal stability, with only the stylus exposed to the sample 34.

For a general version, the NPM has the probe 10 and a z-positioner with the x - y movement provided separately.

Referring to Figure 3, the probe 10 of the preferred form of NPM is connected to a z-positioner 30 which can be a commercial piezoelectric translator. The probe has the force actuator 32 which is the magnet/coil actuator 18, 20 of Figure 1, capacitive sensors 25 for measurement of the normal force/height and frictional force, and stylus 12 with a Berkovich tip for interacting with the sample surface 34. The sample surface 34 is, in turn, moved in x and y directions in an x - y plane by an x - y stage 36 which is controlled through two channels of a DAC (Digital to Analogue Converter) 38 by a control circuit 39.

There are two operational modes for the measurements of height or profile, frictional force and elastic modulus of the sample surface 34. One mode is a general scanning mode for measurements of profile and friction. In this mode a constant force $\boldsymbol{F}$ along the z axis towards the sample set by the combination of the force actuator 32 and the deflection of the beam 16 is applied to the stylus 12. The second mode of operation is a force ramping mode for elastic modulus/hardness measurement. In this mode, the force applied to the sample surface 34 by the stylus 12 is linearly increased from an initial value to a suitable value and then linearly decreased to the initial value. At each scanning point on the sample surface the two modes are operated sequentially.

In the first mode, the force interaction between the stylus 12 and the sample surface 34 which is caused by the height variation of the sample surface and the lateral force applied to the tip are detected by the height and friction capacitive sensors 25. The height signal from the sensors 25 is measured by a capacitive AC bridge 40, applied to an amplifier 42 and active filter amplifier 44, and then to one channel, ADC1, of an Analogue to Digital Converter (ADC) 46. The digitised height signal is then compared with a set or reference value V_{set} in the comparator 47 and an error or difference signal is sent to a control computation unit 48. The output from the control unit 48 is then passed through one channel, DAC1, of a Digital to Analogue Converter (DAC) 50, to a high voltage drive unit 52 which actuates the z-positioner 30 to move the probe in such a way as to maintain the interaction force at the

stylus 12 at the set or reference value i.e. the force F is maintained constant. The output signal from the control unit 48 is an indication of the variation of the height of the sample at the point of contact of the stylus relative to a datum level.

To measure the frictional force, the x - y stage 36 is moved in the x - y plane to "drag" the stylus across the sample surface. The capacitive friction sensors 25 are connected in a differential AC bridge 54 forming a differential capacitive sensor which responds only to the lateral tilt and not the vertical movement of the stylus. The differential AC bridge 54 provides an output signal as a measurement representative of the friction. This is again amplified and filtered by an amplifier 56 and active filter amplifier 58 and then passed to a computer 60 via a second channel ADC2 of the ADC 46 for further processing. The scanning mode is much similar to a general operation of STM (Scanning Tunnelling Probe Microscope)/AFM except that in the NPM the setting force F can be adjusted by the force actuator 32 over a fairly large range up to 10^{-2} N for different applications.

In the force ramping mode, a ramp signal from a ramp signal generator 62 is sent, via DAC2, a second channel of DAC 50, and a current drive 64, to the force actuator 32 to apply an additional force to the sample surface at the stylus 12. This additional force is increased by the ramp signal from the generator 62 which may be increased linearly or in a stepped manner.. The deformation or penetration of the sample surface 34 is monitored by the force/height sensors 25 which monitor changes in the vertical position of the stylus relative to the probe body. The signal from the force/height sensors 25 is applied through the AC bridge 40, amplifier 42, filter/amplifier 44. and ADC1, to the comparator 47, whose output applies a compensating or error signal through the control unit 48 and high voltage drive unit 52 to the z-positioner 30. As a result, the probe is moved in the z axis towards or away from the sample surface in order to maintain the deflection of the beam 16 due to the application of the force F at its initial value. Thus, the penetration or deformation of the sample surface is determined only by the change in F as a result of the application of the ramp signal and the contribution of the beam deflection to the force applied to the stylus is reduced to negligible proportions or eliminated.

The output signal from the control computation unit (or the comparator 47) is a measure of the deformation or penetration and thus the hardness of the sample surface.

However, in the force ramping mode it is possible to switch off the feedback. With there being no feedback control signal to the z-positioner 30, the force applied to the stylus 12 and thus the sample surface is produced by a combination of the beam deflection and the force actuator 32. This mode of operation is suitable for the application of small forces. The output signal from the bridge 40, the amplifier 42, the filter 44 and the ADC 46 is a measure of the deformation/penetration.

The procedure for measuring the height and elastic modulus/hardness of the sample surface 34 at each point and the frictional force is illustrated in the form of a flow chart in Figure 4. On beginning the measurement, the system of NPM is initialised by moving the probe to a preselected position above the sample surface. A preselected signal is applied to the current drive 64 to apply a constant current to the coils 20 of the force actuator 32 and apply the constant setting force $\boldsymbol{F}$ to the stylus 12. The scanning or movement range of the x - y stage, the ramping mode (with or without feedback) and the value and range of the ramping signal is also programmed into the microprocessor 60. A preselected set or reference value is applied to the comparator 47 to generate an error signal which is used to activate the drive unit 52 and thus the z-positioner 30. The latter moves the probe and thus the stylus towards and into contact with the sample surface at the scanning location or point. The z position 30 continues to move the probe towards the surface, applying force to the stylus until the error signal is reduced to zero or a selected level. The setting force $\boldsymbol{F}$ is thus applied by the stylus to the sample surface.

The output signal V_c of the control unit 48 represents the vertical position of the probe (and thus the height of the sample surface at that location) and is here set as a datum level. Changes in this signal as the stylus is repositioned at other locations on the sample surface can be monitored and stored as a relative height measurement in the microprocessor 60 to provide a map of the surface profile or topography. The results can be displayed on the display 70.

The ramping signal V_1 is then applied by the ramp signal generator 62 to the force actuator 32 to apply a ramping force to the sample surface 34 by the stylus 12.

If the feedback loop is switched off then the signal V_p generated through the bridge 40, amplifier 42, filter/amplifier 44 and ADC1 is stored by the microprocessor, together with the ramping voltage V_1. These signals are processed and displayed by the display 70 to give an indication of the elastic modulus/hardness of the sample at the scanning location.

If the feedback loop is on then the output signal of the height capacitive sensor is applied through the bridge 40, amplifier 42, filter/amplifier 44 and ADC1 to the comparator 47 for comparison with the set value and the height of the probe is adjusted by the z-positioner 30 in accordance with the comparison. The height of the probe will continue to be adjusted by the z-positioner 30 until V_P is equal to the set value V_{set}. Then the control signal V_c for the z-positioner 30 and the ramping signal V_1 are stored and processed by the microprocessor 60 for display on the display 70.

The x - y stage is also moved in a preselected direction at a set speed in the x - y plane with the effect that the stylus 12 is "dragged" across the sample surface 34. The twisting of the beam 16 and thus the deflection of the stylus 12 is measured by the differential bridge 54 of the frictional force measurement circuit which generates a signal V_F representative of the measured frictional force. This is stored by the microprocessor 60 and can be displayed on the display 70.

The probe is moved to successive scanning points on the sample surface and the above described measurements are repeated at each scanning point to build up maps of the properties across the sample surface. These include a topography map, frictional force map and hardness/Young's modulus map.

It is also possible to use the force ramping mode to measure adhesion of the sample surface and also shear stress.

In the normal course of a measurement process, the adhesion and shear stress would be measured before measurement of the hardness of the sample surface.

To measure adhesion, the probe and thus the stylus 12 is moved by the z-positioner relative to the sample surface along the z axis towards the sample surface as described above whilst the force applied to the stylus is monitored. The momentary deflection of the stylus caused by contact with the surface is detected. The position of the probe at this contact point is registered as a datum representing the surface and the probe is then withdrawn by the z-positioner 30. The adhesion or resistance to withdrawal of the stylus from the surface is measured by the height sensors 25 as the beam 16 deflects. The level of the output signal from the height sensors is an indication of the magnitude of the adhesion at the surface.

The stylus can be biassed towards the sample surface by the force actuator 32 before contacting the surface or can be held at a neutral position relative to the body by the beam 16 in the absence of any force applied by the force actuator 32.

Once contact is detected, the feedback loop can be arranged to cause the z-positioner 30 to withdraw the probe until the stylus is again at its preselected bias position or at its neutral position relative to the body whilst maintaining contact. At this point $V_p = V_{set}$ and the error signal has returned to zero or its original value.

In order to measure the shear stress, a small oscillation signal is applied either to the probe or to the sample or to both to cause the stylus to oscillate or "dither" in the x - y plane on the surface of the sample. The deflection of the stylus in x - y is in the nanometer level typically between 1 and 10 nanometers. The deflection of the stylus in the x - y plane is measured by the frictional force measurement circuit to provide an indication of the shear stress.

It is possible to use the NPM as an end-point detector to measure surface adhesion forces and static attraction forces over a range of materials. In addition, the NPM can be used as a micro/nano engraving tool to scratch a surface following a required pattern.

It will be appreciated that whilst the probe is described as being moved in the x-y plane and z axis, the movement is relative to the sample and the probe could be held stationary whilst the sample is moved, or a combination of both.

CLAIMS

1 A scanning probe microscope for measuring the hardness of a surface of a sample object at a location on said surface, comprising:

a stage means (36) for mounting the object;

a probe mounted relative to said stage means;

first actuator means (30) for moving the probe and the stage means (36) relative to one another along a first, z axis and in an x - y plane orthogonal to said first, z axis;

wherein the probe has:

a body;

stylus means (12) for contacting the object surface;

mounting means (16) resiliently mounting the stylus to the body so as to allow movement of the stylus relative to the body along the z axis;

second actuator means (32) for moving the stylus means relative to the body along the z axis towards the stage means (36);

and position monitoring means (25) for monitoring the position of the stylus (12) relative to the body along the z axis and providing a position signal representative thereof;

and wherein the microscope further comprises:

a control circuit comprising:

first drive means (52) for actuating said first actuator means (30);

second drive means (50, 60, 62, 64) for actuating said second actuator means (32) to apply a preselected force to the stylus means (12) subsequent to said initial contact,

a feedback circuit connecting the first drive means (52) and the position monitoring means (25) and operable for controlling the first actuator means (30) to bring the stylus (12) and the probe into a preselected relationship along the z axis in response to initial contact of the stylus with the object surface;

and processor means (40, 42, 44, 46, 47, 48, 60) for receiving said position signal and providing a hardness signal representative of the local hardness of the object surface at the point of contact of the stylus (12) with the surface.

2 A scanning probe microscope as claimed in claim 1 wherein said processor means (40, 42, 44, 46, 47, 48, 60) provides said hardness signal in response to movement of the stylus away from said preselected relationship as a result of application of said preselected force to said stylus at said point of contact.

3 A scanning probe microscope as claimed in claim 1 wherein:

said control circuit is operable for controlling the first actuator means (30) to maintain the stylus (12) and the probe in said preselected relationship along the z axis during application of said force at said point of contact;

and said processor means (40, 42, 44, 46, 47, 48, 60) is operable to provide said hardness signal in response to movement of the probe by said first actuator means (30) to return the probe and the stylus to said preselected relationship following movement of the stylus as a result of application of said force.

4 A scanning probe microscope as claimed in any of the preceding claims wherein said

control circuit is further operable to provide a datum representative of the position of the probe relative to said stage (36) at said initial point of contact and to provide a topography signal representative of variations in the height of the object surface at successive points of contact of the stylus (12) with the surface.

5 A scanning probe microscope as claimed in any of the preceding claims wherein said position monitoring means (25) comprises position sensor means formed between said mounting means (16) and said body and having a position parameter varying in dependence on the position of said stylus relative to said body.

6 A scanning probe microscope as claimed in claim5 wherein said position monitoring means (25) comprises capacitative sensor means formed between said mounting means (16) and said body and said parameter is capacitance.

7 A scanning probe microscope as claimed in claim 5 or 6 wherein said control circuit further comprises processor means (40, 42, 44, 50, 47, 48, 60) for providing a position signal in dependence on the parameter of said position sensor means and processing said position signal and providing a signal representative of the position of the stylus relative to said body.

8 A scanning probe microscope as claimed in claim 7 wherein said processor means (40, 42, 44, 50, 47, 48, 60) is operable to process said position signal and provide a signal representative of the hardness of the object surface at the point of contact of the stylus with the object surface.

9 A scanning probe microscope as claimed in claim 8 further comprising store means (60) for storing said topography signals and generating a topography map of the surface of the object.

10 A scanning probe microscope as claimed in claim 4 and any of claims 5 to 9 when appendant to claim 4 wherein said control circuit is operable to compare said position signal at each point of contact of the stylus with the object surface with said datum and provide said

topography signal in dependance thereon.

11 A scanning probe microscope as claimed in claim 4 or any of claims 5 to 10 when appendant to claim 4 further comprising store means (60) for storing said topography signals and generating a topography map of the surface of the object.

12 A scanning probe microscope as claimed in any of the preceding claims further comprising tilt monitoring means (25) for monitoring the tilt of the stylus (12) relative to the z axis during movement of the probe relative to the stage (36) in the x - y plane, and providing a tilt signal in dependence on the amplitude of the tilt, the tilt signal being representative of the frictional force between the object surface and the stylus.

13 A scanning probe microscope as claimed in claim 12 wherein said tilt monitoring means (25) comprises tilt sensor means formed between said mounting means (16) and said body and having a tilt parameter varying in dependence on the tilt of said stylus.

14 A scanning probe microscope as claimed in claim 13 wherein said tilt monitoring means (25) comprises capacitative sensor means formed between said mounting means (16) and said body and said tilt parameter is capacitance.

15 A scanning probe microscope as claimed in claim 13 or 14 wherein said control circuit further comprises processor means (54, 56, 58, 46, 60) for providing a tilt signal in dependence on the parameter of said tilt sensor means and processing said tilt signal and providing a signal representative of the friction of the object surface.

16 A scanning probe microscope as claimed in claim 15 further comprising store means (60) for storing said friction signals and generating a friction map of the surface of the object.

17 A scanning probe microscope as claimed in any of the preceding claims wherein said preselected force is maintained substantially constant during movement of said probe in said x - y plane.

18 A scanning probe microscope as claimed in any of the preceding claims wherein said preselected force is selected in the range 10^{-8} N to 10^{-2} N.

19 A scanning probe microscope as claimed in any of the preceding claims wherein the second drive means (50, 60, 62, 64) is operable to apply a varying force to the stylus means (12).

20 A scanning probe microscope as claimed in claim 19 wherein the varying force increases in a stepped manner.

21 A scanning probe microscope as claimed in claim 19 wherein the varying force increases linearly.

22 A scanning probe microscope as claimed in any of the preceding claims wherein said second actuator means (32) is an electromagnetic actuator.

23 A scanning probe microscope as claimed in any of the preceding claims wherein the second actuator means (32) comprise a permanent magnet (18) mounted on one of the stylus and the body and a coil mounted on the other and adapted to carry an electric current whereby the actuator acts directly on the stylus.

24 A scanning probe microscope as claimed in any of the preceding claims wherein said mounting means (16)comprises a resilient beam means (16a-16d).

25 A scanning probe microscope as claimed in claim 24 wherein said resilient beam means (16) is formed by a plurality of leaf spring arms (16a-16d).

26 A scanning probe microscope as claimed in claim 25 wherein four leaf spring arms (16a-16d) are provided, forming a flexible cross-shaped spring beam.

27 A scanning probe microscope as claimed in claim 25 or 26 wherein said leaf spring

arms (16a-16d) have a thickness of between 25μ and 50μ.

28 A scanning probe microscope as claimed in claim 25, 26 or 27 wherein the body has a plurality of electrodes (22) corresponding in number to the leaf spring arms (16a-16d) and affording a corresponding plurality of capacitative sensors (25).

29 A scanning probe microscope as claimed in any of claims 24 to 28 wherein the resilient beam means (16a-16d) is formed of Copper-Beryllium.

30 A scanning probe microscope as claimed in any of claims 24 to 29 wherein the resilient beam means (16a-16d) forms a common electrode (26).

31 A scanning probe microscope as claimed in any of claims 24 to 29 wherein the common electrode (26) is mounted on the resilient beam means (16a-16d).

32 A scanning probe microscope as claimed in claim 31 wherein the common electrode (26) is a thin silicon wafer.

33 A scanning probe microscope as claimed in claim 28 or any of claims 29 to 32 when appendant to claim 28 wherein the capacitative sensors (25) comprise a pair of capacitative sensors forming said position monitoring means (25) and adapted to measure movement of the stylus (12) in the z axis.

34 A scanning probe microscope as claimed in claim 6 and claim 28 or any of claims 29 to 33 when appendant to claim 28 wherein the capacitative sensors (25) comprise a pair of capacitative sensors forming said tilt monitoring means (25), said capacitative sensors being operated in a differential mode and adapted to measure torsional movement of the stylus mount and hence frictional force at the stylus.

35 A scanning probe microscope as claimed in any of the preceding claims wherein the stylus means (12) has a generally symmetrical mount within the body.

36 A scanning probe microscope as claimed in any of the preceding claims wherein the stylus means (12) is formed from a silica rod.

37 A scanning probe microscope as claimed in any of the preceding claims wherein the stylus means (12) has a diamond tip.

38 A method of measuring the local hardness of an object surface using a scanning probe microscope having a probe comprising:

a body;

stylus means (12) for contacting the object surface;

mounting means (16) resiliently mounting the stylus to the body so as to allow movement of the stylus relative to the body along the z axis;

actuator means (32) for moving the stylus means relative to the body along the z axis towards the stage means (36);

and position monitoring means (25) for monitoring the position of the stylus (12) relative to the body along the z axis and providing a position signal representative thereof;

wherein the method comprises the steps of:

a) monitoring the position of the stylus relative to the probe body in the z axis;

b) moving the probe towards the surface along the z axis until a change in the position of the stylus relative to the body indicates contact of the stylus at a selected point on the surface;

c) moving the probe along the z axis relative to the stylus whilst maintaining contact of the stylus with the object surface until a preselected relationship is established between the positions of the stylus and the body along the z axis to set a datum position for the stylus at said selected contact point;

d) increasing by a preselected amount the force applied to the stylus along the z axis to move the stylus into the object surface;

e) monitoring the movement of the stylus in the z direction relative to the datum and generating a position signal in dependence thereon;

f) and processing said position signal to provide a hardness signal representative of the hardness of the object surface at said selected surface contact point.

39 A method as claimed in claim 38 wherein the position of said probe is maintained constant on the z axis during step d).

40 A method as claimed in claim 38 wherein the step of processing said position signal comprises:

g) during step d) providing a control signal in dependence on said position signal to adjust the position of said probe on the z axis to maintain said preselected relationship, and processing said control signal to provide a hardness signal representative of the hardness of the object surface at said selected surface contact point.

41 A method as claimed in any of claims 38 to 40 comprising repeating the steps a) to f) at a plurality of selected points on the object surface and providing a map of the hardness of the object surface.

42 A method as claimed in claim 41 further comprising:

providing a signal representative of the datum at each said selected contact point on the object surface to provide a plurality of height signals;

and processing said height signals to provide a map of the topography of the object surface.

43 A method as claimed in any of claims 38 to 42 further comprising the steps of:

h) moving the probe relative to the object surface in an x - y plane orthogonal to the z axis whilst maintaining said preselected relationship and maintaining contact of the stylus with the object surface;

i) monitoring the tilt of the stylus relative to the z axis during the movement and generating a tilt signal representative of the degree of tilt;

j) and processing said tilt signal to provide a friction signal representation of the frictional force of the surface.

44 A scanning probe microscope as claimed in any of claims 38 to 43 further comprising the step of:

k) prior to step b) actuating said actuator means to apply a preselected force to said stylus to bias said stylus towards said object surface and establish said preselected relationship between the positions of the body and the stylus along the z axis.

45 A scanning probe microscope as claimed in any of claims 38 to 43 wherein said stylus is biassed in a neutral position relative to said body on the z axis when in said preselected relationship.

46 A scanning probe microscope substantially as hereinbefore described with reference to and as illustrated in the accompanying drawings.

47 A method of measuring the topography, local hardness and frictional force of an object surface using a scanning probe microscope substantially as hereinbefore described with reference to and as illustrated in the accompanying drawings.

FIG 1

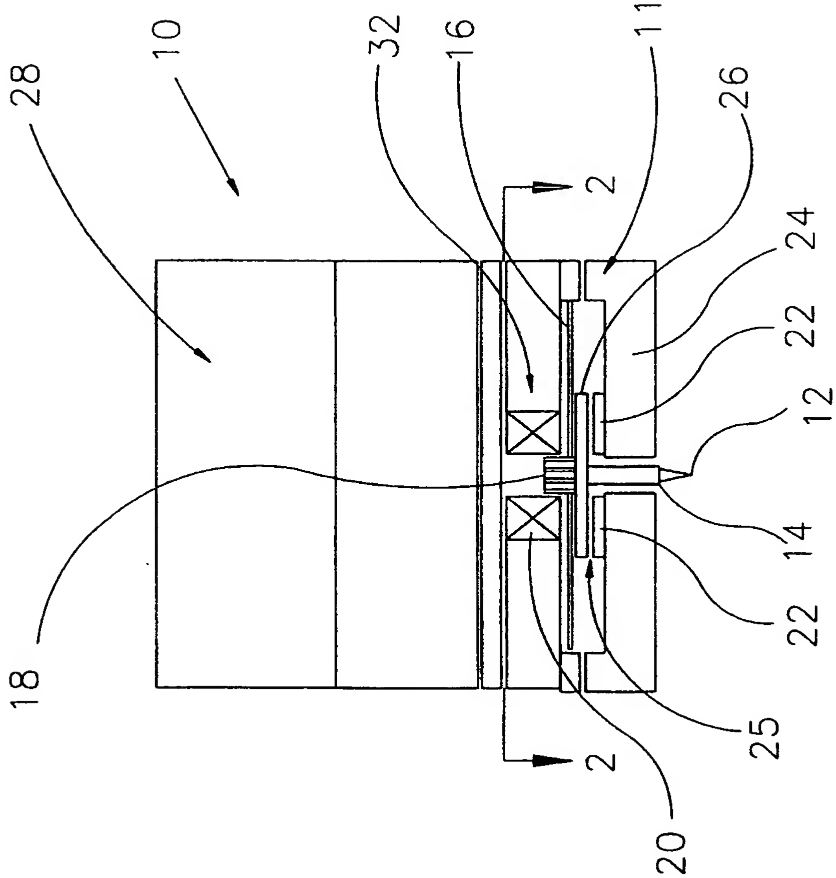
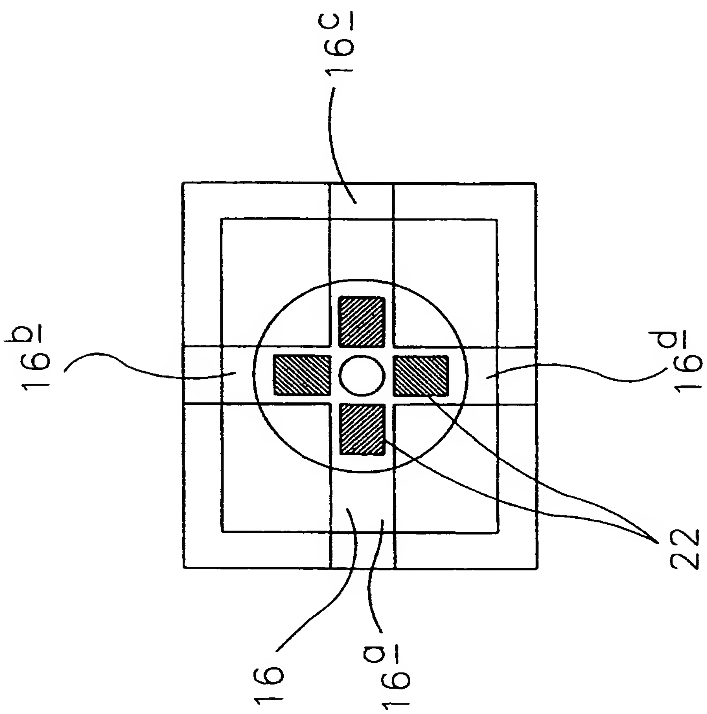
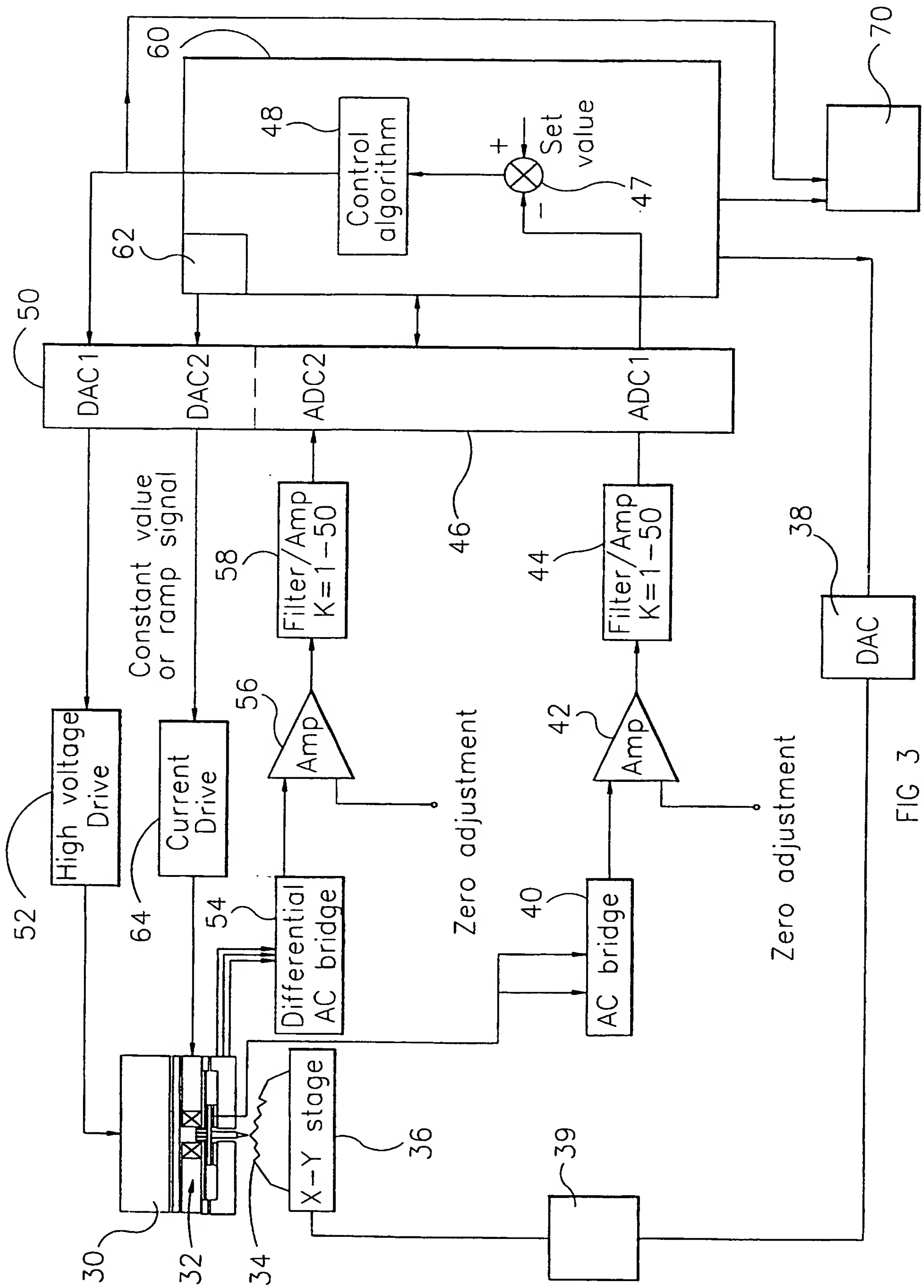


FIG 2





60
70
48
Control algorithm
+
Set value
47
-
62
50
DAC1
DAC2
ADC2
ADC1
46
Constant value or ramp signal
58
Filter/Amp K=1-50
44
Filter/Amp K=1-50
38
DAC
56
Amp
42
Amp
Zero adjustment
Zero adjustment
High voltage Drive
Current Drive
52
64
54
Differential AC bridge
40
AC bridge
X-Y stage
36
39
30
32
34

FIG 3

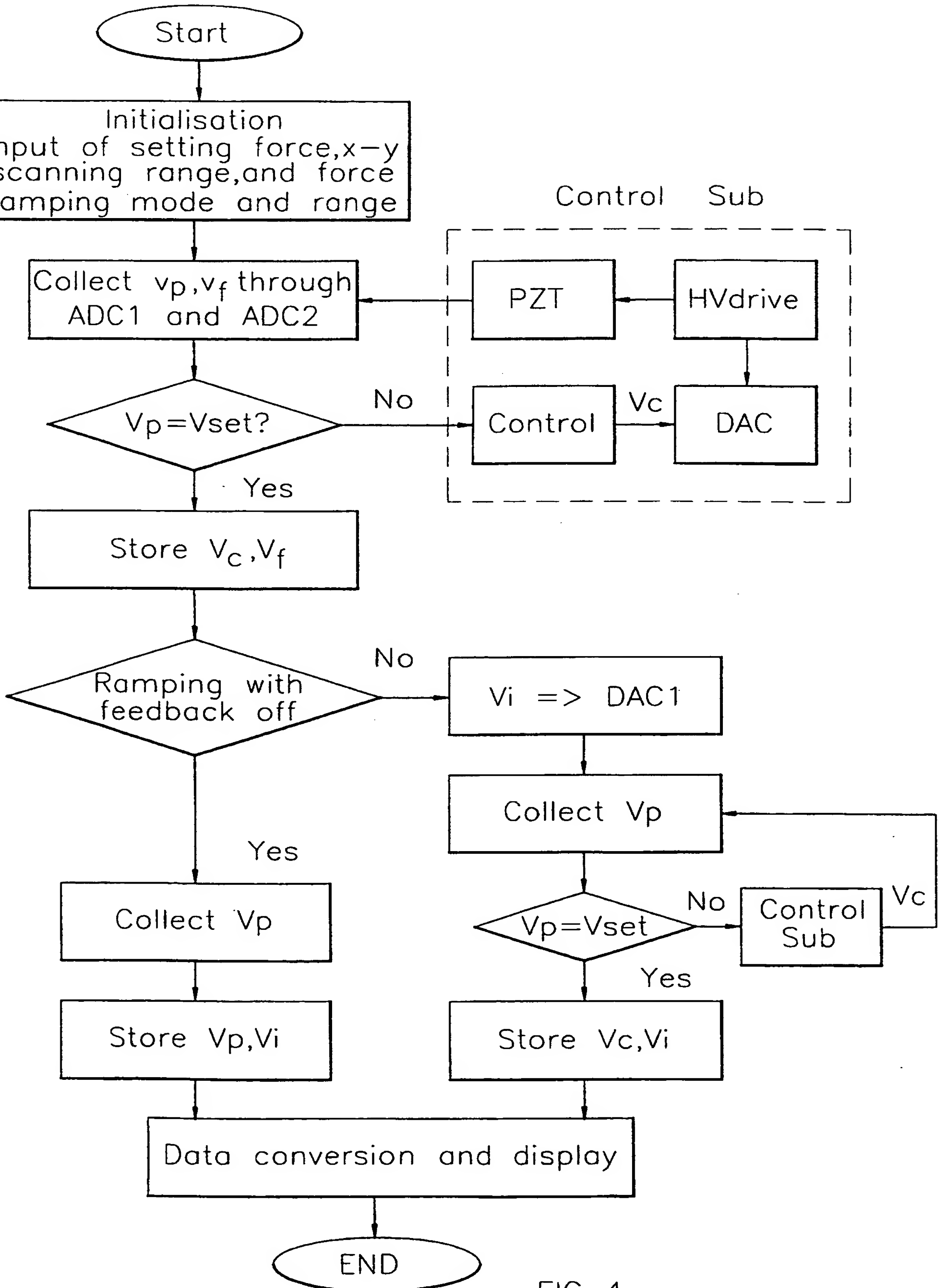

Start
Initialisation
Input of setting force,x−y scanning range,and force ramping mode and range
Control Sub
Collect vp,vf through ADC1 and ADC2
PZT
HVdrive
No
Vp=Vset?
Control
Vc
DAC
Yes
Store Vc,Vf
No
Ramping with feedback off
Vi => DAC1
Collect Vp
Yes
Collect Vp
No
Vp=Vset
Control Sub
Vc
Yes
Store Vp,Vi
Store Vc,Vi
Data conversion and display
END

FIG 4

INTERNATIONAL SEARCH REPORT

International Application No PCT/GB 99/00673

A. CLASSIFICATION OF SUBJECT MATTER

G 01 N 3/42

According to International Patent Classification (IPC) or to both national classification and IPC 6

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

G 01 N,G 01 B

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	EP 0600452 A1 (DIGITAL INSTR., INC.) 08 June 1994 (08.06.94), abstract, column 8, lines 32-50, claims, fig. 1-6.	1,3,7, 9,11
A	WO 96/12930 A1 (HYSITRON INC.) 02 May 1996 (02.05.96), abstract, page 23, line 11 - page 26, line 17, claims, fig. 2,2A,2B,2C.	1,2,3, 5,7,8, 47
A	WO 96/03641 A1 (KLEY, V.B.) 08 February 1996 (08.02.96), abstract, page 6, line 22 - page 7, line 28, claims, fig. 1,11,16,18.	1-9, 22,23

[X] Further documents are listed in the continuation of box C.

[ ] Patent family members are listed in annex.

* Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

Date of the actual completion of the international search

18 May 1999

Date of mailing of the international search report

16. 06. 99

Name and mailing address of the ISA

European Patent Office, P.B. 5818 Patentlaan 2
NL - 2280 HV Rijswijk
Tel. (+ 31-70) 340-2040, Tx. 31 651 epo nl,
Fax (+ 31-70) 340-3016

Authorized officer

ERBER e.h.

INTERNATIONAL SEARCH REPORT

International Application No
PCT/GB 99/00673

-2-

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	Patent Abstracts of Japan, Vol. 97, No. 7, 31 July 1997; & JP,A,09-072925 (NIKON CORP) 18 March 1997, abstract.	1-9

ANHANG | ANNEX | ANNEXE

zum internationalen Recherchenbericht über die internationale Patentanmeldung Nr.

to the International Search Report to the International Patent Application No.

au rapport de recherche international relatif à la demande de brevet international n°

PCT/GB 99/00673 SAE 225865

In diesem Anhang sind die Mitglieder der Patentfamilien der im obengenannten internationalen Recherchenbericht angeführten Patentdokumente angegeben. Diese Angaben dienen nur zur Unterrichtung und erfolgen ohne Gewähr.

This Annex lists the patent family members relating to the patent documents cited in the above-mentioned international search report. The Office is in no way liable for these particulars which are given merely for the purpose of information.

La présente annexe indique les membres de la famille de brevets relatifs aux documents de brevets cités dans le rapport de recherche international visée ci-dessus. Les reseignements fournis sont donnés à titre indicatif et n'engagent pas la responsibilité de l'Office.

Im Recherchenbericht angeführtes Patentdokument Patent document cited in search report Document de brevet cité dans le rapport de recherche		Datum der Veröffentlichung Publication date Date de publication	Mitglied(er) der Patentfamilie Patent family member(s) Membre(s) de la famille de brevets			Datum der Veröffentlichung Publication date Date de publication
EP A1	600452	08-06-1994	DE	C0	69320920	15-10-1998
			DE	T2	69320920	28-01-1999
			EP	B1	600452	09-09-1998
			JP	A2	6213910	05-08-1994
			JP	B2	2837083	14-12-1998
			US	A	5308974	03-05-1994
			US	B1	5308974	06-01-1998
			US	A	5418363	23-05-1995
			US	B1	5418363	06-01-1998
WO A1	9612930	02-05-1996	AU	A1	40095/95	15-05-1996
			EP	A1	805946	12-11-1997
			EP	A4	805946	11-11-1998
			JP	T2	10511176	27-10-1998
			US	A	5553486	10-09-1996
			US	A	5576483	19-11-1996
			US	A	5661235	26-08-1997
			US	A	5869751	09-02-1999
			AU	A1	79636/94	01-05-1995
			EP	A1	721564	17-07-1996
			EP	A4	721564	11-11-1998
			JP	T2	9503864	15-04-1997
			WO	A1	9510021	13-04-1995
			AU	A1	41024/96	06-06-1996
			EP	A1	792437	03-09-1997
			EP	A4	792437	18-11-1998
			JP	T2	10508944	02-09-1998
			WO	A1	9615423	23-05-1996
			EP	A1	868644	07-10-1998
			WO	A1	9722845	26-06-1997
WO A1	9603641	08-02-1996	AU	A1	31527/95	22-02-1996
			JP	T2	10506457	23-06-1998
JP A2	9072925	18-03-1997	keine - none - rien			